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COLUMBIUM AND TANTALUM ALLOY DEVELOPMENT

By Herbert R. Babitzke, Laurance L. Oden, and Hal J. Kelly

• • • • • • report of investigations 7211



UNITED STATES DEPARTMENT OF THE INTERIOR Stewart L. Udall, Secretary

BUREAU OF MINES
John F. O'Leary, Director

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COLUMBIUM AND TANTALUM ALLOY DEVELOPMENT

Herbert R. Babitzke, ¹ Laurance L. Oden, ¹ and Hal J. Kelly²

ABSTRACT

high-temperature structural applications, the Bureau of Mines applied solid solution and precipitation-hardening techniques to columbium and tantalum alloys. Thirty-three alloys were evaluated to determine their formability, As part of a project to develop refractory metal alloys suitable for strength, and oxidation resistance. Three alloys had tensile strengths near 40,000 psi at 1,200° C: CD-IM-3W-5V-10HE (No. 4), CD-1SHE-5W-0.5B (No. 31), and CD-1SHE-5W-1B (No. 32). Oxidation resistance of the high-strength alloys was good. Alloy 4 gained only 34 mg/cm² at 1,200° C, and 21 mg/cm² at 1,000° C in 2 hours. But forming was done without any protection from oxidation.

1 INTRODUCTION

section and because resources of columbium-bearing minerals are more extensive oxidation resistance of columbium and increased its high-temperature strength. Columbium-base alloys rank among the most promising materials for use at temperatures of 1,100° to 1,370° C with high strength-to-weight ratios. Research and development programs on alloys for high-temperature service have favored than those of tantalum. Although there are indications that tantalum alloys are useful above 1,300° C, long life at these high temperatures will depend on the development of suitable oxidation-resistant coatings. For all these columbium over tantalum because of its lower density and lower neutron cross Alloying research by industry and the Bureau of Mines has improved the reasons, more emphasis has been placed on research with columbium than with tantalus. Much research has been conducted for improving columbium and tantalum alloys over the past 20 years. Some of the alloys developed have found industrial application; however, none have the desired combination of strength,

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oxidation resistance, and formability necessary for high-temperature applications. The strength of columbium and tantalum has been incroved by alloying for both solid solution strengthening and precipitation hardening, but many of the resulting alloys cannot be fabricated. For example, alloy F-48 (Cb-15W-5Mb-1Zr, or in atomic percent, Cb-8W-5Mo-1Zr), is one of the strongest alloys but has poor oxidation resistance and is difficult to fabricate. Other alloys have high strength and good formability, but for use at high temperatures the oxidation resistance is far from adequate.

This Bureau of Mines investigation was conducted to determine the effect of several alloying additions on the oxidation resistance and the strength of columbium and tantalum at high temperatures. The results expand data previously collected and published by the Bureau of Mines $(1-7,\ 18,\ 21)^3$ and by such firms as Battelle Memorial Institute $(8\cdot10,\ 13\cdot17)$, Wright Air Development Center (11), Westinghouse Electric Corp. (12), and Wah Chang Corp. $(19\cdot20)$. Alloys developed at the Bureau of Mines during this investigation compare favorably with those made previously and are equivalent in strength to those developed by previous investigators.

PREPARATION OF ALLOYS

Experimental alloys were prepared by adding minor elements to columbium and tantalum. Analyses of columbium and tantalum, purchased as electron-beammelted stock, are shown in table 1. The alloys were prepared first as three 50-gram buttons by arc-melting in a helium atmosphere furnace with a nonconsumable, tungsten electrode. These small buttons were melted three times and then consolidated into one larger button, I inch wide by 3 inches long, by melting twice more. Microscopic examination of sections cut from the buttons revealed no massive segregation.

TABLE 1. - Impurity analyses of columbium and tantalum, ppm

El ement	Columbium	Tantalum	Element	Columbium	Tantalum
Aluminum	07.>	17 - 20	Manganese	<20	_
Boron	₽	⊽	Molybdenum	02>	ot>
Cadmitte	\$>	7	Mickel	0Z>	01>
Carbon	08 - 02>	8	Mitrogen	25- 30	20- 25
Chromitum	87	0 T>	Oxygen	60- 70	95
Cobalt	01>	۵	Silicon	95	017
Columbium	•	S	Tentalum	×500	•
Copper	04>	2 - 2	Tin	<10	01>
Hafnium	8	£	Tit anium	07>	01>
Hydrogen	2.1- 2.3	1.7- 2.0	Tungsten	250- 270	<10
Iron	S	<15	Vanadium	<20	o1>
Lead	87	♡	Zinc	(1)	01×
Magnesium	0Z>	<10	Zirconium	<250	8

Underlined numbers in parentheses refer to items in the list of references at the end of this report.

To add certain elements to the columbium alloys, master alloys were prepared with the following compositions: columbium with 30 weight-percent tungsten, columbium with 4 weight-percent borron, columbium with 14.3 weightpercent carbon, and columbium with 2.4 and 2.9 weight-percent nitrogen. Adding tungsten, boron, carbon, and nitrogen in this way resulted in less segregation than when small amounts of these elements were added directly to the columbium. For the tantalum alloys all the elements were added directly, as were the

The alloys prepared are shown in table 2. To facilitate discussion, the alloys are grouped as follows: Group I alloys were prepared to determine the effects of aluminum, carbon, chromium, copper, hafnium, iron, nitrogen, sill-con, titanium, tungsten, vanadium, and zirconium on the strength and oxidation resistance of columbium. Group 2 comprises alloys in which zirconium was subtituted for the hafnium in the Cb-15Hf-5W alloy to compare results with the two elements. Group 3 alloys are based upon Cb-15Hf-5W with additions of carbon, nitrogen, and boron. Group 4 comprises tantalum alloys. The columbium-iron alloy button of group I shattered during cooling, and was consequently dropped from the investigation. Group 3 alloys were prepared in duplicate.

TABLE 2. - Alloys prepared for this investigation1

(Alloy composition in atomic percent)

- Paramon	TOTA TROPING	Ch. 1 SH f. SW	Ch. 15Hf. 5W-0 10	Cb-15Hf-5W-0.5C 7	Cb-15Hf-5W-1.0C	Cb-15H£-5W-0,1N	Cb-15Hf-5W-0,5N	Cb-15Hf-5W-1,0N	Cb-15Hf-5W-0.18	Cb-15Hf-5W-0 SR 7	Ch. 158f. 50.1 08	90.1-40-1101-00						1	Te-SW-SW-	Te-15Hf. su-28-	and and and	9	
Allov	Group 3:	1342	: :		26									,	<u>۔</u>			Group 4:		17.			
Composition	(20 でんじゅ スト	Cb-1N-5W-3V-5H£	Cb-1N-5W-5V-5HF	Cb-1N-5W-3V-10H£	CP-1N-5W-5V_10HE	C5-1N-5W-3SI-5Hf B	Cb-IN-5W-3Cr-5H£	Co-15Hf-5W-4S1 P	Cb-15Hf-5W-4S1-1N E	Cb-15Hf-5W-2S1,-2A, F Cb	Cb-15Hf-5W-2Zf-4A]-1CA	Cb-15Hf-5W-2Zr-1C #	Cb-15Hf-2Zr-4V-1C.	Cb-10T1-4A1-0.1Cu-2W	Cb-14.6IE-3.9Zr-13.3HF-3.5A1-0.7NK	Cb-14.8T1-3.9Zr-13.4Hf-3.4S1 L	Cb-69Fe_3M	٥	Cb-5Zr-5W		Cb-152r-5W	Cb-20Zr-5W 1/h	Cb-33Zr-5W
Alloy	Group 1:	1	2					,		9	10	11	12	13	14	15	18	Group 2:	19	20	21	22	23

Nominal values are used throughout the paper.

Data from sample 13a correspond to a sample in reference 6, p. 22.

EVALUATION OF ALLOYS

Fabrication

Sections from all of the buttons were hammer-forged at room temperature to achieve a SO-percent reduction in overall thickness. For those alloys that cracked upon cold forging, another section from the same button was hammer-forged at 1,200°C. The information obtained was used as a guide in forging the remaining portion of the button. A smaller reduction was made for the bot-forged specimens than for the cold-forged specimens because it was necessary to grind the hot-forged specimens to remove surface contamination.

con additions. However, the alloys containing silicon and no tungsten could be hot-forged. By forging was also required for the columbium-zirconium-tungsten alloys in group 2, whereas all the alloys in group 3 could be cold-forged. Compared with the results of the Ch-Hf-W alloys studied in a previous investigation (4), the rhenium addition to the tantalum alloy in group 4 reduced ductility. Table 3 shows that the fabricability of group 1 alloys was decreased most by the sili-

TABLE 2

Alloy	Alloy composition,	Reduction,	percent		æ	Results1	C1
	atomic percent	Room temp.	1,200° C Poor	Poor	Fair	Poog	Fair Good Excellent
Group 1:				L			
1	Cb-11f-54f-3V-58ff	47	31	×	,	,	>
2	Cb-111-5W-5V-SH£	65	38	H	,	>	٠,
3	Cb-114-5W-3V-108ff	52	*	H	,	٠,	>
4	Cb-1st-5w-5v-108f	39	27	×	٠	,	` >
2	CP-18-54-3S1-3Ef	45	3	À	'	,	٠, ١
••••	Cb-114-54-3Cr-5Hf	53	31	, ×	•	,	>
7	Cb-15HE-5W-4S1	8	(e)	, 2	,		٠,
8	C9-15HE-5W-4S1-1H.	5	(e)	ì }			•
6	Cb-15H£-5W-251-2A1	7	(3)	ì }			,
10	Cb-15Hf-5H-2Zr-4A1-1C	7,	È	ì×	,		> >
1	Cb-15Hf-5W-2Zr-1C	53			٠	,	•
12	Cb-158f-2Zr-4V-1C	. 57	,	_		,	
13	Cb 1071-4A1-0.1Cu-2W	: 5	,			•	•
16	Cb-14,6Ti-3.9Zr-13.78f-3.5Al-0.7m	: 9	. ;		• 1		×
	Ch-14.8Ti-3.92r-13.48f-3.454	3	7 76	•	H	,	^
		3	Ŗ	×	,		>
19	O-52r-54	ð	36				
8		; ;	8 8		×		>
-	O-157-60	2.5	8 8		×	>	
	- 13/2 - 34	۸:	25	,	×	,	*
	CB-202r-54	25	23	,	×	,	Α.
23	CP-332r-24.	41	33	,	×		. >
Group 3:4							•
134	Cb-158£-5¥	51,54	•	,	,	×	*
24	Cb-158f-54-0.1C	45.54	,	,	,	×	. >
25	CB-15Hf-5W-0.5C	49-55	,	,	,	×	i #
26	CB-158£-54-1.0C	05-97	•	ı	•	×	
27	Cb-15Ef-5E-0.1K	49-51	•	,	,	×	*
28	Cb-15HE-5W-0.5M	48-55		,	,	×	۱ ،
29	Cb-15Hf-5W-1.0M	52-55		,	•	×	,
30	Cb-15H£-5W-0.1B	51-54	,	,			,
31	Cb-154f-54-0.58	54,55	,	,	•	×	*
32	Cb-154f-54-1.08	50.57	,	,	,		٠,
Group 4:							•
91	Ta-54-546	3			,	,	×
17 Ta-1 946 - 54-28e	To. 1 Caf. St. 22	;	7		_		

cracks, but some usable mate-*roor-many cracks, no usable material available; fair-many cracks, but rial available; good--few edge cracks; excellent--no cracks.

*x-forging results at room temperature; y--forging results at 1,200° C.

*Failed at 1,500° C.

*Results are for duplicate alloys.

All alloys that could be forged were cleaned by etching in a solution of hydrofluoric and nitric acids and heat-treated for 5 hours in vacuum at 1,300° C. Previous experience with columbium alloys showed that recrystallization occurred with this combination of time and temperature. If the alloys were not sufficiently ductile to be cold- or hot-forged, no further tests were performed.

Rolling to a final thickness of 0.06 inch was done either at room temperature or at 1,000° C with 5 percent reductions each rolling pass. Samples for hot rolling were preheated 5 minutes. No protection from oxidation was provided.

The alloys in group I could not be rolled if silicon had been added. In group 2 only the alloys that were low in zirconium content were rolled successfully. All the alloys in group 3 were ductile to the extent that they could be cold rolled with only limited edge cracking. The tantalum-hafinium alloy containing both tungaten and rhenium was not sufficiently ductile to be rolled into sheet. The rolling data are shown in table 4.

TABLE 4. - Columbium and tantalum alloy rolling results

CD-1N-5W-3V-5HE Room temperature G7 Good	Allov	Alloy composition	Bo114-c		
CD-1N-5W-3V-5HE Room temperature 67 CD-1N-5W-3V-5HE Room temperature 67 70 CD-1N-5W-5V-5HE Room temperature 67 70 70 70 70 70 70 70 70 70 70 70 70 70		atomic nercent	tomperature	Reduction,	Kesults
CD-1N-5N-3V-3HF Goom temperature 67 CD-1N-5N-9V-3HF Godo 771 CD-1N-5N-9V-10HF Godo 771 CD-1N-5N-9V-10HF Room temperature 56 CD-1N-5N-3C-5HF Room temperature 63 CD-1NH-5N-2C-5HF Room temperature 63 CD-13HF-5N-2C-4N-1C Room temperature 65 CD-13HF-5N-2C-4N-1C Room temperature 65 CD-13HF-2C-4A-1-C Room temperature 65 CD-13HF-2C-4A-1-C Room temperature 65 CD-13HF-3N-2C-13-3HF-3.5A-10-7N Godo 66 CD-15HF-3N-3C-13-3HF-3.5A-10-7N Godo 66 CD-15H-5N-1-C Room temperature 60,73 CD-15H-5N-1-C Room temperature 60,73 CD-15H-5N-1-C Room temperature 60,73 CD-15H-5N-1-C Room temperature 67,68 CD-15H-5N-1-C Room tem	Group 1:		a incertadana	חבורבוור	
CD-1N-5N-5V-5HF do. 70 CD-1N-5N-7V-10HF do. 70 CD-1N-5N-7V-10HF do. 70 CD-1N-5N-3Si-5HF Room temperature 56 CD-1SHE-5N-2Zr-4Al-1C Room temperature 74 CD-1SHE-5N-13-3Zr-13-3HF-3.5Al-0.7N do. 66 CD-1CT-4Al-0.1Ql-2N-3Sh-0.7N do. 66 CD-1SZr-5N- Room temperature 76 CD-1SHE-5N-0.1C Room temperature 77 CD-1SHE-5N-0.1C Room temperature 60,73 CD-1SHE-5N-0.1C Room temperature 61,73 CD-1SHE-5N-0.1C R	1	Cb-1N-5W-3V-5H£	Room temperature	.,	7
CD-1N-5W-3V-10HE Good resperature SG GO-1N-5W-3V-10HE Room temperature SG GO-1N-5W-3C-5HE Room temperature G1 GOOO C. GD-1SHE-5W-2C-5HE Room temperature G1 GOOO C. GD-1SHE-5W-2C-5HE Room temperature G1 CD-1SHE-2X-4N-1C. Room temperature G1 S9 GO-1SHE-2X-4N-1C. Room temperature G1 S9 GO-1SHE-2X-4N-1C. Room temperature G1 S9 GO-1SHE-3W-2C-13-3HE-3-5A1-0.7N G-0-1SHE-3W-3C-13-3HE-3-5A1-0.7N G-0-1SHE-3W-3C-13-3HE-3-3HE-3-3HE-3-3HE-3-3HE-3HE-3HE-3H	2	Cb-1N-5W-5V-5Hf	www.cemperature	6 5	. Doog
CD-1N-5M-5S-59-10HE Good temperature 56 CD-1N-5M-3S-54-6HE Room temperature 56 CD-1SHE 5W-2Zr-641-1C. 1,000° C. 59 CD-1SHE 5W-2Zr-641-1C. 1,000° C. 59 CD-1SHE 5W-2Zr-641-1C. 1,000° C. 59 CD-1SHE 5W-2Zr-1O10-2W. Room temperature 65 CD-1SHE 3W-2Zr-13-4HE 3.5A1.0.7N Go. 60 CD-1SZ-5W. 100° C. 60 CD-1SHE 5W-0.1C. 100° C. 60 CD-1SHE 5W-0.0C. 100° C. 60 CD-1SHE 5W-0.		Ch-1 N-5U-3V-10HF		7 .	· on
CD-18-35-37-10ff CD-18-37-37-10ff CD-18-37-37-10ff CD-18-37-37-10ff CD-18-37-37-30ff CD-18-37-37-37 CD-18-37-37-37 CD-18-37-37-37 CD-18-37-37-37 CD-18-37-37-37 CD-18-37-37 CD-18-37 CD-37 CD	,	_		2	2
Color 1,000 Color 1,000 Color Colo	:		op	19	Do.
CD-1M-5M-3Cr-5Hf Room temperature 63 CD-1SHf-5N-2Zr-4Al-1C 1,000° C. CD-1SHf-2X-2Zr-4Al-1C 1,000° C. CD-1SHf-2X-2Zr-4Al-1C 1,000° C. CD-1SHf-2X-13.9Zr-13.3Hf-3.5Al-0.7N do 66 CD-14.6Tl-3.9Zr-13.3Hf-3.5Al-0.7N do 66 CD-15Zr-5W 1,000° C 60 CD-15Hf-5W-0.1C 60 CD-15H		CD-1N-2N-3S1-5HE	Room temperature	ж	Fair.
CD-18-58-05-54ff Room temperature 63 CD-18-58-05-27-4A1-1C Room temperature 59 CD-15Hf-5W-2Zr-1C Room temperature 59 CD-15Hf-5W-2Zr-1C Room temperature 65 CD-15Hf-2Xr-1C Room temperature 65 CD-15Hf-2Xr-13-44ff-3.451 Room temperature 69 CD-15HF-31-32r-13.3Hf-3.5A1-0.7N Room temperature 60 CD-15Gr-5W Room temperature 60 CD-15Gr-5W Room temperature 60 CD-15Gr-5W Room temperature 60 CD-15Gr-5W Room temperature 60 CD-15HF-5W-0.1C Room temperature 60			and 1,000° C.		
CD-15HE-SW-2Zr-4Al-1C. CD-15HE-SW-2Zr-4Al-1C. CD-15HE-ZZr-4V-1C. CD-15HE-ZZr-4V-1C. CD-10T1-4Al-0.1Gu-2W. CD-14.6Tl-3-9Zr-13.3HE-3.5Al-0.7N. CD-14.6Tl-3-9Zr-13.4HE-3.4Sl. CD-15Zr-5W. CD-15Zr-5W. CD-15Zr-5W. CD-15Zr-5W. CD-15Zr-5W. CD-15Zr-5W. CD-15HE-SW. CD-	9	Cb-1N-5W-3Cr-5Hf	Room temperature	63	Good.
CD-15HE-5H-2Zr-1C Room temperature 74 CD-15HE-2Zr-4Al-0.10a.2W do 65 CD-14.6Ti-3.9Zr-13.3HE-3.5Al-0.7N do 66 CD-14.6Ti-3.9Zr-13.4HE-3.4Si do 66 CD-5Zr-5W do 770 CD-15Zr-5W Room temperature 76 CD-15Zr-5W Room temperature 69 CD-15HE-5W-0.1C do 770 CD-15HE-5W-0.1C do 69,71 CD-15HE-5W-0.1C do 65,72 CD-15HE-5W-0.1C do 65,72 CD-15HE-5W-0.1C do 65,72 CD-15HE-5W-0.1C do 770 CD-15HE	10	Cb-15Hf-5W-2Zr-4A1-1C	1,000° C	59	Excellent.
CD-15HE 2Zr-4V-1C, do 65 CD-14.6T1-4A1.010-2L, do 56 CD-14.6T1-3.3HE-3.5A1.0.7N CD-14.6T1-3.9Zr-13.4HE-3.4S1, do 69 CD-16.2Zr-5W CD-10Zr-5W CD-10Zr-5W CD-15Zr-5W CD-15Zr-5W CD-15Zr-5W CD-15HE-5W CD-	11	_	Room temperature	74	Good
CD-14.61.0.1 Cu. 2W. do 66 CD-14.611.3.9Zr-13.3Hr-3.5A-0.7N do 66 CD-14.611.3.9Zr-13.3Hr-3.4Si. do 66 CD-14.611.3.9Zr-13.3Hr-3.4Si. do 69 CD-15Zr-5W. do 70 CD-15Zr-5W. 1,000° C 60 CD-15Hr-5W. 1,000°	12	_	do		Freellent
CD-14.6T1-3.92r-13.3Hf-3.5Ai-0.7N CD-12r-5W CD-12r-5W CD-12r-5W CD-15r-5W CD-15r-5W CD-15r-5W CD-15HF-5W-0.1C	13		Q.	3	Per center
Cb-15.87 - 3.92r-13.4Hf-3.4S1	14	_	ą.	3 3	3 7
CD-5Zr-5W. 1,000° C. 100° C. 1	15			9 5	
CD-5ZE-5W. -00. CD-10Zr-5W. 1,000° C. CD-15ZZ-5W. 1,000° C. CD-15ZZ-5W. 1,000° C. CD-15HE-5W. 69 CD-15HE-5W. 67 CD-15HE-5W. 1,000° C. Ta-5W-5W. 1,000° C. Ta-15HE-5W-2Re. 1,000° C. Ta-15HE-5W-2Re. 1,000° C. Ta-15HE-5W-2Re. 1,000° C. Ta-15HE-5W-2Re. 1,000° C.		***************************************		ŝ	œ.
CD-10ZZ-5W, 1,000°C, 60 CD-10ZZ-5W, 1,000°C, 60 CD-10ZZ-5W, 1,000°C, 60 CD-15ME-5W-0.1C, 69 CD-15HE-5W-0.1C, 60 CD-15HE-5W-0.1	oroup 2:	;			
CD-10Zz-5W. 1,000° C 60 CD-15Zz-5W. 1,000° C 60 CD-15Zz-5W. 1,000° C 76 CD-15HE-5W. Room temperature 60,73 CD-15HE-5W. 60,73 67,68 CD-15HE-5W. 72,73 72,73 CD-15HE-5W. 72,73 72,73 CD-15HE-5W. 70,71 72,73 CD-15HE-5W. 70,71 70,71 CD-15HE-5W. 70,71 70,71 CD-15HE-5W. 70,59 66,72 CD-15HE-5W. 70,71 66,72 CD-15HE-5W. 70,05 66,72 CD-15HE-5W. 70,00 66,72 CD-15HE-5W. 70,00 66,72 CD-15HE-5W. 70,00 66,72 CD-15HE-5W. 70,00 65,68 CD-15HE-5W. 70,00 <td>19</td> <td>Cb-5Zr-5W</td> <td>qo</td> <td>۶</td> <td>Excellent.</td>	19	Cb-5Zr-5W	qo	۶	Excellent.
CD-15ZZ-5W. Room temperature 76 CD-20ZZ-5W. 1,000° C. 69 CD-15HE-5W. 60,73 60,73 CD-15HE-5W. 60,73 60,73 CD-15HE-5W. 72,73 72,73 CD-15HE-5W. 70,71 70,71 CD-15HE-5W. 70,71 70,71 CD-15HE-5W. 70,71 70,71 CD-15HE-5W. 70,60 67,69 CD-15HE-5W. 70 67,69	20	Cb-10Zr-5W	1,000° C	09	ъ.
Cb-15HE-5W. 1,000° C. 69 Cb-15HE-5W. Room temperature 60,73 Cb-15HE-5W-0.1C. do. do. 77,73 Cb-15HE-5W-0.5C. do. 72,73 Cb-15HE-5W-1.0C. do. 70,71 Cb-15HE-5W-0.1N. do. 67,71 Cb-15HE-5W-0.1N. do. 67,71 Cb-15HE-5W-0.1B. do. 67,71 Cb-15HE-5W-0.1B. do. 66,71 Cb-15HE-5W-0.1B. do. 65,71 Cb-15HE-5W-0.1B. do. 65,71 Cb-15HE-5W-0.1B. do. 65,68 Cb-15HE-5W-0.5B. do. 62,68 Cb-15HE-5W-0.5B. do. 63,67 Ta-5W-5Wo. 1,000° C. 53,67	21		Room temperature	92	Poor.
CD-15HE-5W. CD-15HE-5W-0.1C. CD-15HE-5W-	22	_	1.000° C	69	Center opened un
CD-15HE-5W. CD-15HE-5W-0.1C. 60.0m temperature 60.73 CD-15HE-5W-0.1C. do 72.73 CD-15HE-5W-0.1C. do 70,71 CD-15HE-5W-0.1N. do 67,69 CD-15HE-5W-0.1N. do 67,69 CD-15HE-5W-0.1N. do 69,71 CD-15HE-5W-0.1N. do 66,72 CD-15HE-5W-0.1N. do 65,71 CD-15HE-5W-0.1N. do 65,71 CD-15HE-5W-0.1N. do 65,71 CD-15HE-5W-0.1N. do 53,67 Ta-5W-5Wo. 1,000° C. 57 Ta-15HE-5W-2Re. 1,000° C. 53	Group 3:2	_			.45
Cb. 18HE-SH-0.1G do 67,68 Cb. 18HE-SH-0.5G do 72,73 Cb. 18HE-SH-10G 70,71 70,71 Cb. 18HE-SH-0.1N do 71,73 Cb. 18HE-SH-0.10 do 67,69 Cb. 18HE-SH-0.10 do 66,72 Cb. 18HE-SH-0.5B do 66,72 Cb. 18HE-SH-1.0B do 63,67 Ta-5W-5MO 1,000° C 57 Ta-18HE-SH-2Re 1,000° C 53	13a	_	Room temperature	60.73	Poor
Cb-15HE-5H-0.5C db-15H-273 Cb-15HE-5H-10C db-172,73 Cb-15HE-5H-0.1N db-171,73 Cb-15HE-5H-0.5N db-171,73 Cb-15HE-5H-0.5N db-171,73 Cb-15HE-5H-0.5B db-172,73 Cb-15HE-5H-0.5B db-172,73 Cb-15HE-5H-0.5B db-172,73 Cb-15HE-5H-1.0B db-172,73 Cb-15HE-5H-5H-0.5B db-172,73 Cb-15HE-5H-5H-0.5B db-172,73 Cb-15H-5H-5H-0.5B db-172,73 Cb-15H-5H-5H-0.5B db-172,73 Cb-15H-5H-5H-0.5B db-172,73 Cb-15H-5H-0.5B db-172,73 Cb-15H-0.5H-0.5B db-172,73 Cb-15H-0.5B db-172,73 Cb	24	Cb-15Hf-5W-0.1C	do	67.68	Excellent
CD-15HE-5W-1.0C, do 70,71 CD-15HE-5W-0.1N, do 71,73 CD-15HE-5W-0.5N, do 67,59 CD-15HE-5W-1.0N, do 69,71 CD-15HE-5W-0.1B, do 66,72 CD-15HE-5W-0.1B, do 66,72 CD-15HE-5W-1.0B, do 63,67 Ta-5W-5W0, do 63,67 Ta-5W-5W0, do 63,67 Ta-15HE-5W-2Re, do 63,67 Ta-15HE-5W-5W0, do 63,67 Ta-15HE-5W0, do 63,	25	Cb-15Hf-5W-0.5C	do.	27. 73	-
Cb-15HE-5H-0.1N. -do 71,73 Cb-15HE-5H-0.5N. -do 67,69 Cb-15HE-5H-0.1B. -do 69,71 Cb-15HE-5H-0.1B. -do 62,72 Cb-15HE-5H-0.5B. -do 62,68 Cb-15HE-5H-1.0B. -do 63,67 Ta-5H-5Ho. -do 57 Ta-15HE-5H-2Re. 1,000° C. 57	26	Cb-15Hf-5W-1.0C,	do	70,71	8
CD-15HE-5W-0.5N. 67,69 CD-15HE-5W-1.0N. 60 CD-15HE-5W-0.1B. 66,72 CD-15HE-5W-0.5B. 66,72 CD-15HE-5W-0.5B. 66,72 CD-15HE-5W-0.5B. 65,68 CD-15HE-5W-0.5B. 63,67 Ta-5W-5Wo. 71,000 C 57	27	Cb-15Hf-5W-0,1N,	do	27.73	2
CD-15HE-5M-1.0N. do 69,71 CD-15HE-5W-0.1B. do 66,72 CD-15HE-5W-0.1B. do 62,72 CD-15HE-5W-1.0B. do 62,68 CD-15HE-5W-1.0B. do 63,67 Ta-5W-5W0. do 57 Ta-15HE-5W-2Re. 1,000 C 57	28	Cb-15Hf-5W-0.5N	do	67,69	100
CD-15HE-5H-0.1Bdo 66,72 CD-15HE-5H-0.5Bdo 62,68 CD-15HE-5H-1.0Bdo 63,67 Ta-5H-5HOdo 53,67 Ta-5H-5HOdo 57	29	Cb-15Hf-Sw-1.0N	do	69.71	į
Cb-15Hf-5W-0.5Bdo 62,68 Cb-15Hf-5W-1.0Bdo 63,67 Ta-5W-5Wodo 57 Ta-15Hf-5W-2Re 57	30.	Cb-15Hf-5W-0.1B.	9	66, 73	Freellent
Cb-15Hf-5W-1.0B. 63,67 Ta-5W-5Wo. 63,67 Ta-15Hf-5W-2Re. 57 Ta-15Hf-5W-2Re. 53 Fe	31	Cb-15Hf-5W-0.5B.	C.	62,68	De lette
Ta-5W-5Wo. 57 Ta-15M£-5W-2Ma. 57 Ta-15M£-5W-2Ma. 53 Fa	32	Cb-158f-5W-1.08.	ę.	2010	i a
Ta-5W-5Wo. 57 Ta-15Mf-5W-2Re. 53 Fe	Group 4:			10,00	ġ
Ta-15Hf-5W-2Re 1,000° C 53 Fa	16	Ta-5W-5Wo.	ť	C	į
33	17	Ta-15Hf-5W-2Re	7 0000	3 2	3
			1,000	2	Fair.

Excellent.-no cracks; good--few edge cracks; fair--many cracks but some usable mererial available; poor--many cracks, no usable material available.
Results are for duplicate samplar.

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Strength

9

Elevated-temperature tensile tests were made at 1,200° and 1,400° C at a strain rate of 0.001 in/in/sec. The specimens were in the annealed condition and measured 0.04 inch thick, 0.250 inch wide, and 5 inches long, with no reduced gage section. Tests were made on a Marquardt tensile testing machine described in a previous report (6). The alloys were electrically heated by self-resistance in a vacuum of 10⁻⁵ torr. A platinum versus platinum-10 percent rhodium thermocouple was spotwelded to the center of the specimen to about 1 minute.

Three of the columbium alloys tested at 1,200° C had tensile strengths near 40,000 psi (see table 5); alloy 4 of group 1 contained Cb-1N-5W-5V-10Hf, and alloys 31 and 32 of group 3 contained Cb-15Hf-5W, plus 0.5 and 1 atomic percent boron, respectively. In addition to solid-solution strengthening, precipitation hardening due to nitride or boride formation probably occurred. No exceptional values were observed for alloys in group 2 and 4. Since only one alloy composition in group 2 was fabricated into tensile specimens, no strength comparisons were made between this group and the Cb-15Hf-5W alloy.

Several low strength values were observed for some of the alloys tested; some showed defects such as inclusions, and some showed defects from fabrication. In several tests the samples failed outside the gage length. In most of these instances, the results were discarded. None of the current 13a (Cb-15Hf-5W) samples gave valid strength results; therefore results from a previous investigation (6) are reported in table 5.

Metallography

Microstructures of some of the alloys are shown in figure 1. They show that complete recrystallization has taken place and that one or more phases are distributed throughout the specimens. Alloy 13a (Cb-15HE-5M) shows a solid-solution structure with a little second phase. The specimens used for metallography were cut from the cold portions of the tensile specimens. The alloys had been recrystallized at 1,300° C for 5 hours in a vacuum of 10-5

Oxidation Behavior

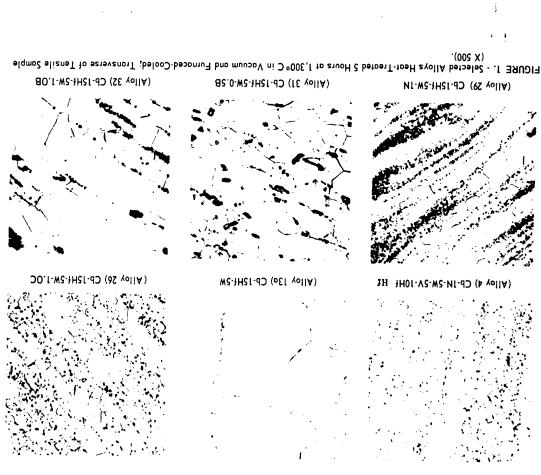
The alloys that rolled successfully were cut into samples measuring approximately 0.1 by 2 by 2 cm, cleaned, and recrystallized. To determine oxidation resistance, the specimens were tested in air for 2 hours at 1,000° and 1,200° C. Testing was done in a vertical tube furnace that was open on top and closed at the bottom. During oxidation testing, the weight of each sample was continuously recorded with an automatic recording balance.

'Reference to specific makes or models of equipment is made to facilitate understanding and does not imply endorsement by the Bureau of Mines.

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					om reference 6, p. 22, used.	ri bibū ^s
Chisel.		1	1		e occurred outside gage length.	*Fractur
Irregular.		30.9	4.55	007 1	Ta-5W-5MC-WZ-BT	16
2011100221	5	38.4	2.24	1,200	Ta-5W-5M2-WZ-BT	91
-	17	25.5	4.72	087'1	CP-12HE-5W	13gs
-	<u> </u>	9.48	2.26	515'1	CP-15HE-5W.	13as
	56	9,7£	0.24	1,200	CP-15H£-5W	138E1
Irregular.	52	4.96	0.04	1,200	Cb-15H£-5W-1B	32
D⊙.	(_T)	9.55	7.68	1,200	Cb-15Hf-5W-1B	32
.od	(_T)	(_T)	€.8€	1,200	Cb-15H£-5W-1B	32
.od	1 5	(+)	0.24	1,200	Cb-15Hf-5W-0.5B	31
Do.	9	8.05	8.75	1,200	Cb-15Hf-5W-0.1B	30
Do.	15	1.25	€.25	002'1	CP-12HE-5W-1C	30
Brittle.	٤٤	32.4	1.25	002,1	CP-15HE-5W-0.5C	
.od	(_T)	9.62	9.0€	1,200		25
Do∙	81	8.15	6.25	1,200	ער זוווע פון טייט פון	52
Do.	12	2.82	8.55	1,200	22 0 113 31131 113	25
Chisel.	17	32.2	6.98	1,200	בר כווס פווס ווס	24
.oa	36	0.02	20.9	002 1	CP-15HE-2M-0.1C	24
Brittle.	(_T)	24.5	2.62	007 1	CP-52r-5W	61
Do.	\ `\£9	2.81	21.4	1,200	113 -23 -10	61
po·	78	2.81	7.02	002 1	Cb-14.6Tt-3.9Zr-13.3Hf-3.5Al-0.7N	14
Do.	έε	6.91	2.02	1,200	Cb-10Tf-4A1-0.1Cu-2W.	13
po·	17	0.25	0.25	1,200	Cb-10Ti-4AI-0.1Cu-2W	ε1
po.	87	24.5			CP-15HE-22r-4v-1C	51
Ďo.	07	34.8	24.5	005 1	CP-15HE-5W-2Zr-4A1-1C	01
Do.	(_T)	0.71	0.25	1,200	CP-15HE-5W-2Zr-4A1-1C.	01
.og	Šĭ	1.05	0.25	1,400	CP-IN-2M-3CF-2HE	9
Do.	16		0.25	1,200	CP-IN-SM-3CF-SHE	9
po·	78	42.1	1,24	1,200	CP-IN-2M-2Λ-IOH€	٠٠٠٠٠ -
Chisel.	7¢ 7¢	0.55	0.88	1,200	CP-IN-SM-3A-IOHE	£
1,40		32.9	6.25	1,200	CP-114-5W-3V-5HE	1
tracture	don't I ni	isq 000,1	1 tag 000,1	, O o		
	percent	(0.2 percent offset)		temperature,	Alloy composition	Sample
Type of	Elongation	Vield strength	Tensile	Testing		
	2/222	10 0	'9)			

TABLE 5. - Elevated-temperature tensile test data for columbium and tantalum alloys



10Hf in group 1) had low rates of oxidation. Alloy 4 also had high strength (42,000 psi) at 1,200° C. Over a 2-hour period the weight gain was 34 mg/cm² at 1,200° C and only 21 mg/cm² at 1,000° C. This is better than a twofold improvement over unalloyed columbium and about equal to alloys developed by other investigators (12). Oxidation resistance improved with increasing zirconium content in group 2 and compared favorably with the Cb-15Hf-5W alloy. ALLOY SAMPLE NUMBERS

24 25 26 27 27 28 29 30 31

FIGURE 2. - Oxidation Data for Columbium and Tantalum Alloys. WEIGHT GAIN, mg/cm

9 2

Group 2 8

Group 3

0

12

5 4 5

As shown in figure 2, all the alloys in group 3 and alloy 4 (Cb-IN-5W-5V-IOHf in group 1) had low rates of oxidation. Alloy 4 also had high strength

CONCLUSIONS

- be increased substantially by combining with small amounts of other elements, The strength and oxidation resistance of columbium and tantalum may while sufficient ductility may be retained to permit fabrication. Of the elements used for alloying, the most effective strengtheners were tungsten, vanadium, hafnium, nitrogen, and boron. Oxidation resistance was also improved.
- Three alloys had tensile strengths of 40,000 psi at 1,200° C. alloy compositions were as follows:

Cb-1N-5W-5V-10Hf (alloy 4) Cb-15Hf-5W-0.5B (alloy 31) Cb-15Hf-5W-1.0B (alloy 32)

- 3. The three strongest alloys also had good resistance to oxidation at $1,000^\circ$ and $1,200^\circ$ C. Better than a twofold improvement over unalloyed columbium was noted, and each alloy was about qual to some of the commercial alloys developed to date.
- 4. The three alloys listed have shown enough merit to warrant further investigation of their engineering properties. In addition a more intensified study is recommended for alloys containing boron additives to the base composi-tion, Cb-ISHF-5W.

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